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THIN-FILM PERMANENT MAGNETS FOR INTEGRATED ELECTROMAGNETIC COMPONENTS

Queens College of CUNY

F. J. Cadieu

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APPROVED: Peter J. Rainville

PETER J. RAINVILLE
Project Engineer

FOR THE COMMANDER: John K. Schindler

JOHN K. SCHINDLER, Director
Electromagnetics & Reliability Directorate

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F. J. Cadieu

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Queens College of CUNY
Physics Department
Flushing NY 11367

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13. ABSTRACT (Maximum 200 words)

Methods were developed under this program that allow the deposition and photo-patterning of relatively thick (up to 100 microns) high-energy product (15-25 MgOe) highly oriented films of Sm-Co based permanent magnet materials onto various substrate materials, including GaAs and sapphire. The deposition process allows the magnetization to be in any direction desired in the plane of the film. The films have a Curie point of 700 Celsius and are stable. NdFeB films, with magnetization perpendicular to the plane of the film, were also deposited. Methods developed included: Direct crystallization, in the presence of a magnetic field, of the sputtered materials onto heated substrates; the use of a Aluminum oxide boundary layer to promote adhesion and allow the growth of thick films; and the use of Ar and Xe as a sputtering gas to thermalize the sputtered atoms. Magnets such as those developed under this contract are a significant advance towards providing thin-film bias magnets for planar nonreciprocal microwave devices such as circulators and isolators.

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1. F. J. Cadieu, H. Hegde, and K. Chen, The Synthesis of $Sm_2(Co,Fe,Zr)_{17}$ High Energy Product, 16 to 30 MGOe, Thick Sputtered Films, Presented at 1989 International Magnetics Conference, Washington, D.C., March 28-31, 1989. IEEE Trans. on Magnetics, MAG-25, 3788 (1989).

2. F. J. Cadieu, H. Hegde, and K. Chen, High Energy Product Sm-Co Based Sputtered Films, Crystal Texturing and Magnetic Properties, 34th Magnetism and Magnetic Materials Conference, Boston, November 1989, J. Appl. Phys. **67**, 4969 (1990).

3. H. Hegde, K. Chen, and F. J. Cadieu, Fe Enriched Sm-Co Based High Energy Product Sputtered Films With Precise Crystal Textures, 35th Magnetism and Magnetic Materials Conference, Paper GA-12, 1990, J. Appl. Phys. **69**, 5850 (1991).

4. K. Chen, H. Hegde, and F. J. Cadieu, Sputter Synthesis of Modulated Multilayer High Energy Product Permanent Magnet Films, 35th Magnetism and Magnetic Materials Conference, Paper HA-12, 1990, J. Appl. Phys. **69**, 6064 (1991).

5. F. J. Cadieu, H. Hegde, and K. Chen, Enhanced Crystal Texture Control For Sm-Co Based Films Sputtered In Ar-Xe Gas Mixtures, Paper presented at 8th International Conference on Thin Films, San Diego, April 1990, Thin Solid Films **193/194**, 857 (1990).

6. H. Hegde, Sputter Synthesis of Sm-Co Type Magnetic Films With Strong Alignment of Easy Axes of Grains, Ph. D. Thesis, Physics Department, Queens College of CUNY and the Graduate Center of CUNY, April 1990.

7. F.J. Cadieu, "Permanent Magnet Thin Films" in Physics of Thin Films, Vol. 16, Academic Press, NY (in press) 1991.

Scientific Personnel

Professor F. J. Cadieu, Principal Investigator, Department of Physics, Queens College of CUNY, Flushing, NY 11367.

Dr. Hari Hegde, Dr. Hegde was supported as a Ph.D. graduate student of the City University under this contract, and most recently has received partial support as a Postdoctoral Research Associate. He received the Ph.D. degree at the May 1990 commencement of CUNY.

Mr. Kailai Chen has been supported as a City University of New York Ph.D. candidate under this contract. Mr. Chen has had his thesis proposal approved and should be completing his Ph.D. thesis during the Spring 1992 Semester.

THIN FILM PERMANENT MAGNET FILMS FOR INTEGRATED ELECTROMAGNETIC COMPONENTS

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Abstract

The crucial most important research advance of this program was the development of methods that allow relatively thick high energy product films of Sm-Co based permanent magnet materials to be directly crystallized onto heated substrates during the sputter deposition. It has been necessary to directly crystallize the sputtered films so that the easy axes of the crystallites can be aligned with a specific orientation with respect to the plane of the substrates. This was necessary to optimize the magnetic properties and magnetic energy density that could be retained by the films. A boundary layer of Al-Al_2O_3 formed by presputtering Al in an oxygen rich RF discharge has been successfully used to allow the formation of strongly adherent films with thicknesses at least the order of 50 μm. A boundary layer thickness of 0.25 to 0.50 μm has generally been used. The films must be nearly fully dense to allow a high magnetic energy density to be realized. Such films have been stable with time for at least the period of three years. Static energy products of 15 to 25 MGOe as measured at room temperature have been realized for a large number of thick depositions. The principal class of magnetic films deposited in these studies have been rare earth transition metal, Sm-Co based systems. When an in the film plane magnetization was desired, the principal material deposited has been Sm-Co based films crystallizing into the $TbCu_7$ disordered hexagonal 1-5 structure, or directly into the 1-5 hexagonal structure for richer Sm concentrations. The Curie point of this material is at least 700 °C so that electronic components constructed using these films as magnetic circuit elements can be expected to operate stably without cooling over the entire range of military specifications. Such would not be the case for films largely composed of $Nd_2Fe_{14}B$. Films have been deposited with different Sm to transition metal ratios and with different Fe to Co ratios. Films with Sm concentrations ranging from 7 at.% to 25 at.% have been studied. Such transition metal rich compositions have the advantage of being corrosion resistant. In some parallel studies Sm-Fe films have been deposited that exhibit large remanent flux densities perpendicular to the film plane. The use of thermalized sputtering to limit high energy neutral atom bombardment of the films was necessary for the synthesis of films that exhibit high in plane energy products. The use of Ar-Xe mixtures as a sputtering gas significantly aided in the thermalization of Sm and transition metal sputtered atoms. The RE to TM ratio can be varied by varying the total pressure as well as the Xe partial pressure. Recently we have begun to fabricate magnet circuits by photofabrication methods from high energy product magnetic films the order of 35 μm thick. The first devices fabricated were strip transmission lines such that the strip was situated between the poles of a film patterned magnet. Some depositions using GaAs and Si substrates have also been done with satisfactory results. The GaAs and Si substrates were precoated with an Al-Al_2O_3 boundary layer by sputtering Al in an oxygen rich RF discharge.

SIGNIFICANT RESEARCH ADVANCES

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I. THE SYNTHESIS OF ADHERENT THICK SPUTTERED FILMS

The crucial most important research advance of this program was the development of methods that allow relatively thick high energy product films of Sm-Co based permanent magnet materials to be directly crystallized onto heated substrates during the sputter deposition.[1,2,3] This was an important step for several reasons. It has been necessary to directly crystallize the sputtered films so that the easy axes of the crystallites can be aligned with a specific orientation with respect to the plane of the substrates. This is necessary to optimize the magnetic properties and magnetic energy density that can be retained by the films. This requires that the substrate temperature during film growth is sufficiently high that the deposit crystallizes upon deposition. This requires that the substrate temperature be at least the order of 350 °C during deposition. There is sufficient thermal mismatch between the magnetic materials and the substrates used so that if films are made onto bear substrates the films cannot be made to adhere to the substrates for film thicknesses greater than about 12 μm. In a paper reported in the IEEE Transactions on Magnetics we reported on the use of a boundary layer that was deposited onto the substrates before the deposition of the hard permanent magnet material.[1,2] A boundary layer of Al-Al_2O_3 formed by presputtering Al in an oxygen rich RF discharge has been successfully used to allow the formation of strongly adherent films with thicknesses at least the order of 50 μm. A boundary layer thickness of 0.25 to 0.50 μm has generally been used. Figure 1 shows the fractured cross section of a high energy product Sm(Co,Fe,Cu,Zr) film 49 μm thick. The film

was directly crystallized by sputtering onto a boundary layer coated aluminum oxide substrate. The right side of Fig. 1 shows a x10 magnification of the film substrate interface. The boundary layer which is necessary to allow the strong adhesion of such a thick film is evident. It should also be noted that the film does not exhibit a discernible columnar growth morphology that is usually typical of crystalline sputter deposited films. The films must be nearly fully dense to allow a high magnetic energy density to be realized. The maximum magnetic energy density is proportional to the square of the remanent magnetization. Consequently even a small amount of voids in the film which lowers the available magnetization greatly diminishes the magnetic energy density. Such films have been stable with time for at least the period of three years. Static energy products of 15 to 25 MGOe as measured at room temperature have been realized for a large number of thick depositions.

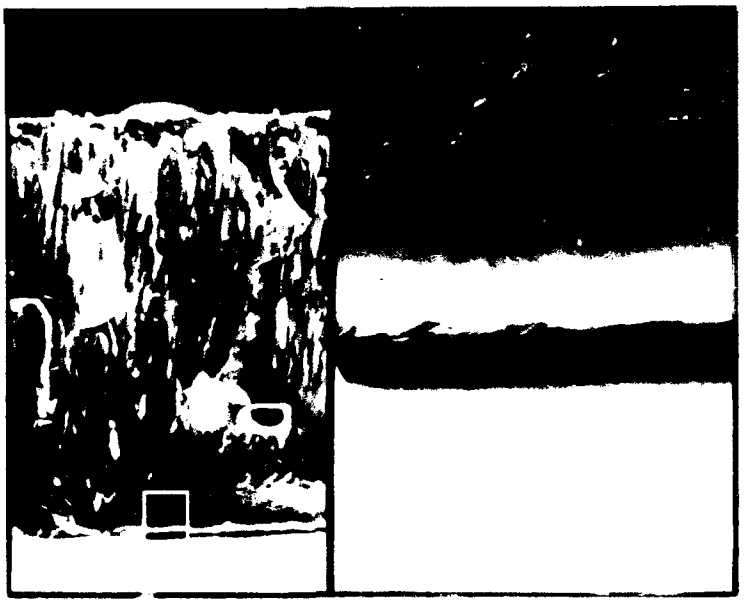

Fig. 1. The cross section of a 49 μm thick Sm(Co,Fe,Cu,Zr) $TbCu_7$ type film that was directly crystallized onto a heated precoated sapphire substrate is shown. The 10X enlargement of the interface region indicated is also shown. Note that the enlargement section exhibits no discernible columnar or void structure. The fracture surface was made

by diamond scribing the back side of the substrate and then breaking the substrate and film.

In some parallel studies Sm-Fe films have been deposited that exhibit large remanent flux densities perpendicular to the film plane. In that case films with magnetizations directed perpendicular to the film plane and static energy products up to 21 MGOe at room temperature have been obtained.[4] These studies involving the synthesis of new iron rich rare earth transition metal permanent magnet systems were supported by the Department of Energy. Once it was shown that it was possible to synthesize these new systems with the crystallite c-axes oriented perpendicular to the film plane, attempts have been made to incorporate such films into permanent magnet film geometries. Some mention of these new perpendicular anisotropy systems synthesized in our laboratory is included in this report for that reason.

The principal class of magnetic films deposited in these studies have been rare earth transition metal, Sm-Co based systems. When an in the film plane magnetization is desired, the principal material deposited has been Sm-Co based films crystallizing into the $TbCu_7$ disordered hexagonal 1-5 structure[2] or directly into the 1-5 hexagonal structure for richer Sm concentrations.[3] This material has been used for several reasons. Firstly the Curie point of this material is at least 700 °C so that electronic components constructed using these films as magnetic circuit elements can be expected to operate stably without cooling over the entire range of military specifications. Such would not be the case for films largely composed of $Nd_2Fe_{14}B$. The ferromagnetic Curie point of $Nd_2Fe_{14}B$ is comparatively low at 330 °C. This has several important consequences. One is that the magnetization of $Nd_2Fe_{14}B$ has a large temperature coefficient at temperatures as low as 100 °C. Another is that the low Curie point of $Nd_2Fe_{14}B$ means that films directly crystallized onto heated substrates are being synthesized above their ferromagnetic Curie point. The films are then

nonmagnetic while being formed so that magnetic anisotropy energies cannot be used to help influence the film growth dynamics which results in specific texturing. Texturing in this case means a preferential alignment of the crystallite easy axes of magnetization. It has not been possible to align the easy axes of $Nd_2Fe_{14}B$ crystallites either perpendicular to the film plane, or onto the film plane, to the same extent as for the different Sm based films with either cobalt or iron.

Secondly, besides having a much higher Curie point, the magnetic properties of the Sm-Co based and Nd-Fe based films are comparable at room temperature. The cost savings due to using Fe based $Nd_2Fe_{14}B$ for bulk applications are not realizable since the processing costs for the film fabrication of electronic components outweighs the material costs. Hence gold films, although thin, are extensively used for microelectronic applications. Since the ferromagnetic Curie point of the Sm-Co based films is generally well above the desired direct crystallization growth temperature, the self demagnetization energy can be used to favor the growth of crystallites with the easy axes of magnetization aligned onto the film plane. The dominant factor determining the in plane normal anisotropy for directly crystallized $TbCu_7$ and $SmCo_5$ type films is due to the demagnetization energy.[5]

Relatively thick films of Sm-Co based permanent magnet films have been deposited onto precoated sapphire and Al_2O_3 substrates. Films have been deposited with different Sm to transition metal ratios and with different Fe to Co ratios. Films with Sm concentrations ranging from 7 at.% to 25 at.% have been studied. Such transition metal rich compositions have the advantage of being quite corrosion resistant. In general the higher Sm concentrations lead to higher as sputtered intrinsic coercivities at the expense of remanent, and saturation magnetizations.

II. FILMS WITH LARGE REMANENT MOMENTS AND HIGH COERCIVITIES

Large remanent moments and high coercivities allow high values of magnetic energy densities to be realized. The most useful measure of the magnetic energy density is the static energy product. The static energy product is the maximum value of B times H internal, H_{int}, in the demagnetizing quadrant of the hysteresis loop. We have predominantly been interested in systems for which energy products of at least 15 MGOe can be obtained at room temperature. High energy product films can be classified by the direction the easy axis of magnetization makes with the film plane.[6,7] Three cases are possible. For case one the easy axis of magnetization lies in the film plane with isotropy in the film plane. For case two, the easy axis of magnetization is in the film plane and at a particular angle within the plane of the film. For case two the use of some agent to induce in-the-film-plane-anisotropy, such as a magnetic field applied parallel to the film plane during film growth and or crystallization, is required. And for case three, the easy axis of magnetization is directed normal to the film plane. In order to control the direction of the easy axis of magnetization it is necessary to preferentially align the easy axes of the individual crystallites. Such film which exhibit a nonrandom orientation of the crystallites are said to be textured. For certain individual compound systems it has been possible to optimize the degree of texturing for in plane easy axes. For other compounds it has been possible to optimize the degree of easy axes alignment perpendicular to the film plane. It has not been possible to vary the sputtering conditions to fully align the same compound system in plane and perpendicular to the plane. However, it has been possible to synthesize $Nd_2Fe_{14}B$ films to exhibit both anisotropies to a large extent by varying the sputtering conditions.[8]

A. High Energy Product Films With In Plane Magnetization

A large number of films have been deposited from commercially available TDK type Sm_2Co_{17} magnet material.[1,2] It has been shown that films with Sm concentrations ranging from 12 to 18 at.% can be deposited by varying the sputtering conditions from these same targets.[3] This has the advantage for subsequent studies and film scale device applications that sputtering target material can be readily obtained without undue expense or difficulty. The films deposited from such targets for properly thermalized sputtering conditions have compositions such as was reported in IEEE Trans. on Magnetics, MAG-25, 3788 (1989).

TDK Sputtering Target Composition

Element	Atomic %
Sm	14.2
Co	55.5
Fe	20.1
Cu	7.8
Zr	2.4
Total	100.0

From such targets films have been deposited so that in plane energy products the order of 15 to 18 MGOe were obtained. The usual thickness range that has been investigated ranges from less than 1000 Å to 100 μm. For films to be strongly adherent and stable, with thicknesses greater than about 12 μm, a boundary layer was required as previously discussed.[1] The usual as sputtered coercivity for such films was approximately 5 to 8 kOe. The usual remnant moment was about 8.3 kG for $4\pi M$. The films that are deposited from a single set of targets could be varied in their Sm to TM metal component ratio by varying the sputtering gas composition and total pressure. Sputtering gas mixtures of Ar-Xe were used to optimally thermalize the sputtered atoms before they reached the substrates. This had the added advantage that higher Xe concentrations in the sputtering gas tended to preferentially scatter the light mass Co and other transition metal atoms relative to the more massive Sm atoms. The Sm atomic percent

collected in the films could be varied from 12 to 18 at.% by varying the Ar-Xe gas ratio and the total sputtering gas pressure.[3] The directly crystallized films sputtered from these targets form in the $TbCu_7$ disordered 1-5 hexagonal crystal structure with a $\approx$ 5 A, and c $\approx$ 4 A.[2] The as deposited films of this type readily develop the maximum magnetic properties. The coercivity and magnetic energy density observed for such films are very little affected by any subsequent heat treatments or annealings.

The film geometry itself also introduces a demagnetization field which favors the growth of films with the easy axes of magnetization aligned onto the film plane. The self demagnetization field can only affect the film texture for systems synthesized below their ferromagnetic Curie point. As an example of these effects, it should be noted that for a random arrangement of uniaxial crystallites in 3-dimensions, the expected ratio between the remanent induction, B_r. and the saturation flux density, $4\pi M_S$, is 0.50. We have demonstrated it is possible to make high energy product films such that the c-axes of all the crystallites are tightly aligned onto the film plane. For a random arrangement of uniaxial crystallites in 2-dimensions, the expected ratio between the remanent induction, B_r, and the saturation flux density is $2/\pi = 0.637$.[5]

It is a new result of these studies that the film composition and consequently the crystal structure can be varied by changing the Ar-Xe partial pressures. Higher Xe partial pressures increase the Sm content of the films with the structure changing from the disordered Sm_2Co_{17}, $TbCu_7$ type structure, to the $SmCo_5$ type structure.

A significant result of these studies is that it has been possible to directly crystallize Sm-Co based films that exhibit very low remanence ratios for hysteresis loops measured out of the plane, to in the plane. Films

with out of plane to in plane remanence ratios as low as 0.04 have been made for films sputtered in Ar-Xe mixtures and Xe. Such a set of hysteresis loops for a film that was sputtered in 60 mTorr Ar50%Xe as the sputtering gas is shown in Fig. 2. These measurements were made on samples as removed from the sputtering system without any subsequent heat treatment. The in the film plane static energy product for the film of Fig. 2 was 17.4 MGOe as measured at room temperature.

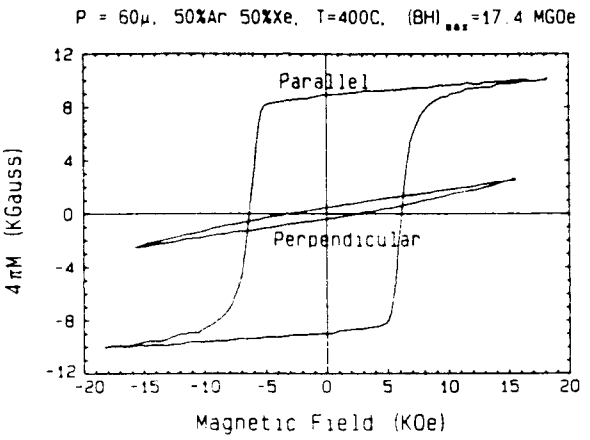

Fig. 2. In-plane and perpendicular to plane hysteresis loops for a 5 μm thick Sm-Co based $TbCu_7$ type structure film sputtered with 60 mTorr Ar 50%Xe at a substrate temperature of 400 °C. These loops were measured at room temperature.

Another significant result is that it has been possible to control the Sm percentage retained in the deposited films by varying the sputtering gas or gas mixture used and the gas partial pressures. These results are a direct result of the different cross sections and energy transfer efficiencies for Ar-Co, Ar-Sm, Xe-Co, and Xe-Sm collisions. Sufficiently high pressures have been used that the neutral sputtering atoms undergo at least several

collisions with the sputtering gas atoms before the substrate is reached. The various similar mass transition metal, TM, atoms in the target, Co, Fe, Cu, Zr, behave similarly so that the main difference arises between the Ar-TM, Ar-Sm, and Xe-TM, Xe-Sm collisions. It should be noted that the Sm atoms are ≈2.5 times as massive as the Co atoms and that the energy transfer efficiency is a function of the mass ratio between that of the sputtered target atoms and that of the sputtering gas atoms.[3] Some other aspects of thermalized sputtering as applied to magnetic materials have been discussed in previous papers.[6,7] Collisions of the less massive TM atoms with the massive Xe atoms also tend to deflect the sputtered TM atoms through larger relative angles than collisions with Ar atoms. A net effect of the differences in the collision properties is that the percentage of the Sm retained onto the substrate can be varied for different gas compositions and pressures. High relative pressures and or partial pressures of Xe results in films that have an increased concentration of Sm. The difference in the Sm content has been analyzed directly on the film pieces measured by electron excited fluorescence in a scanning electron microscope. The substrate temperature was held at the same value of 400 °C for these different sets of depositions. The measured composition differences for films deposited by RF sputtering onto substrates 5 cm distant from the targets with a composition of 14.5 $\pm$0.2 at.% Sm is shown in Fig. 3. Analytic calculations have shown that on the average there are no collisions for pressures of approximately less than 10 mTorr so that little effect would be expected for normal low pressure RF sputtering. In addition to the composition shift effect, the films deposited in Ar-Xe mixtures can be made with better crystal texture control. This is a direct result of the Xe collisions preventing high energy neutral atoms in the sputtered beam from disrupting the crystal phase growth. The

better crystal texture control results in films without any crystallites with c-axes skewed out of the film plane. This then leads to the low ratios for out of plane to in plane remanence ratios as shown in Fig. 2.

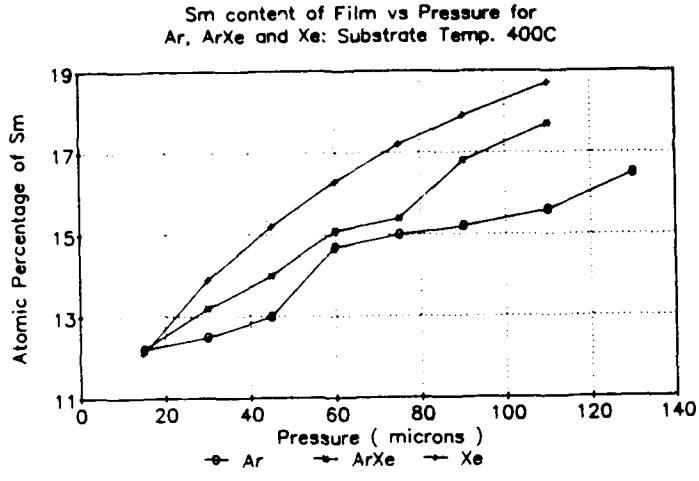

Fig. 3. The variation of the at.% Sm versus sputtering gas pressures for different gas mixtures. All of this data refers to films directly crystallized onto substrates held at 400 °C.

An x-ray diffractometer trace for a film such as that shown in Fig. 2 that was sputtered in 60 mTorr Ar50%Xe is shown in Fig. 4. All of the film diffraction lines from 20 to 90 degrees 2Θ can be indexed as (hk0) lines for the disordered 2-17 phase of the hexagonal $TbCu_7$ type. It should be noted that the y-axis for this figure is a log scale to show the smaller peaks present. The rhombohedral 2-17 phase has similar lines, but has a much higher c/a ratio than the hexagonal form. The short c/a form is consistent with the magnetic properties and is favored by the sputtering conditions.

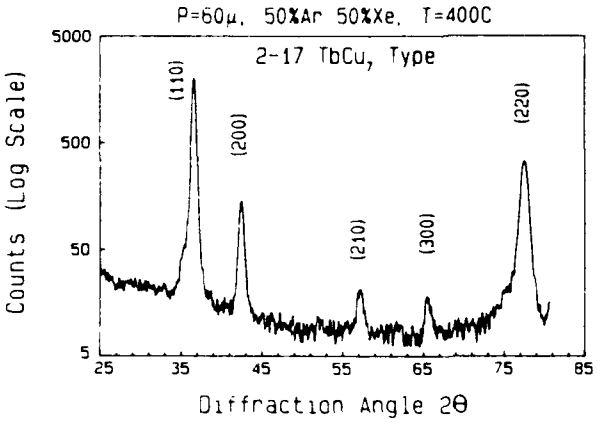

Fig. 4. An x-ray diffractometer trace for the film of Fig. 1 is shown for Cu Kθ radiation. The y-scale is a log scale to show the smaller peaks. The indexing is for the $TbCu_7$ type structure.

As the composition shifts toward richer Sm concentrations, the crystal phasing expected should start to exhibit 1-5 and 2-17 phases at approximately 16.7 at.% Sm. Such a splitting of the lines does start to become pronounced for Ar-Xe pressures as the expected composition becomes greater than 16.0 at.% Sm. Films made at 90 mTorr of Ar still show only the 2-17 phase, but films made at 90 mTorr total pressure but 50% Xe begin to show splitting at 2θ = 36°, 42°, and 77° for $Cu_{K\alpha}$ radiation. Films made with 90 mTorr Xe exhibit predominantly the 1-5 phase with a small shoulder due to the 2-17 phase. The separation of the line at 77-78° into (220) peaks for the 2-17 phase and (220) peaks for the 1-5 phase is shown in Fig. 5. The respective compositions for these films were 15.0 at.% Sm for 90 mTorr Ar, 16.8 at.% Sm for 90 mTorr Ar50%Xe and 17.9 at.% Sm for 90 mTorr Xe sputtered films. The room temperature in plane energy products for these films were: 16.2 MGOe for the film sputtered in 90 mTorr Ar, 14.9 MGOe for the film sputtered in 90 mTorr Ar50%Xe, and 14.6 MGOe for the film sputtered in 90 mTorr Xe. A possible alternative phase boundary would be

that the splitting is between the 1-5 and 2-7 phases. But in that case there is no expected shift for the hexagonal a lattice parameter in going from one phase to the other. The a parameter of the 1-5 phase is slightly larger than the a parameter of the $TbCu_7$ 2-17 phase. The angular shift for the (220) reflection for Cu radiation in going from the 2-17 $TbCu_7$ phase to the 1-5 phase is 2.6° for the pure compositions. The observed angle and composition shifts are consistent with the phase transition being from the $TbCu_7$ form to the 1-5 type structure.

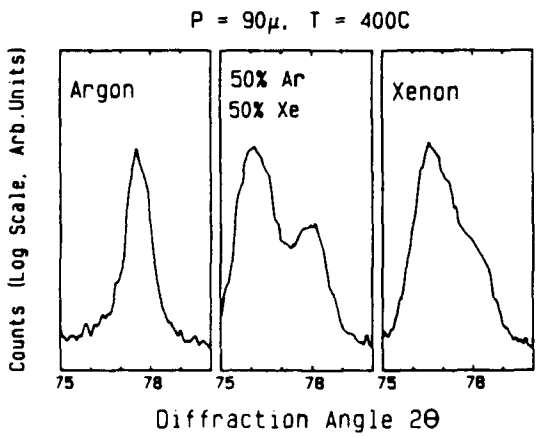

Fig. 5. Parts of x-ray diffractometer traces for films sputtered at 400 °C and 90 mTorr pressure are shown for different gases.

The films compared have been sputtered onto substrates held at the same nominal temperature of 400 °C to show the effects of varying the gas composition on the film at.% Sm. As the partial pressure of Xe is increased higher substrate temperatures can be used to oppose the increased flux of Sm reaching the substrate, but this would lower the overall deposition rate for films of a given composition.

It has been possible to synthesize Sm-Co based films with the $TbCu_7$ type structure that are highly crystallographically aligned with variable compositions by varying the sputtering gas pressure and partial pressures. The use of thermalized sputtering to limit high energy neutral atom bombardment of the films is necessary for the synthesis of films that exhibit high in plane energy products. The use of Xe as a sputtering gas significantly aids in the thermalization of the comparable mass Sm atoms. A lower total pressure of Xe can be used to sputter films that exhibit in plane square hysteresis loops than if Ar only were used.

B. THE SYNTHESIS OF HIGH ENERGY PRODUCT FILMS WITH ANISOTROPY WITHIN THE PLANE OF THE FILMS

The expected degree of in-the-film-plane alignment that a magnetic field applied during the sputter synthesis can induce was discussed in the article titled "Permanent Magnet Thin Films". This book chapter is scheduled to be published in Thin Film Physics, Vol. 16.[5] The model assumes that the growth of crystallites which result in a lower energy state grow preferentially during the direct crystallization of the films onto heated substrates with a magnetic field, H_S, applied in the film plane. After a certain growth thickness, the crystallites tend to grow with a certain growth texture. No epitaxy is necessary, and the textured films can be grown onto polycrystalline substrates. By this sputter process control very thick highly textured films have been grown. The results express the expected ratio of the remanent flux density as a fraction of the saturation flux density. The expected ratio for a collection of uniaxial single domain grains synthesized as a film are shown in Fig. 6. The calculated results are in general

agreement with the experimental results. Mr. Kailai Chen is currently doing a more careful analysis of the experimental results as a part of his thesis.

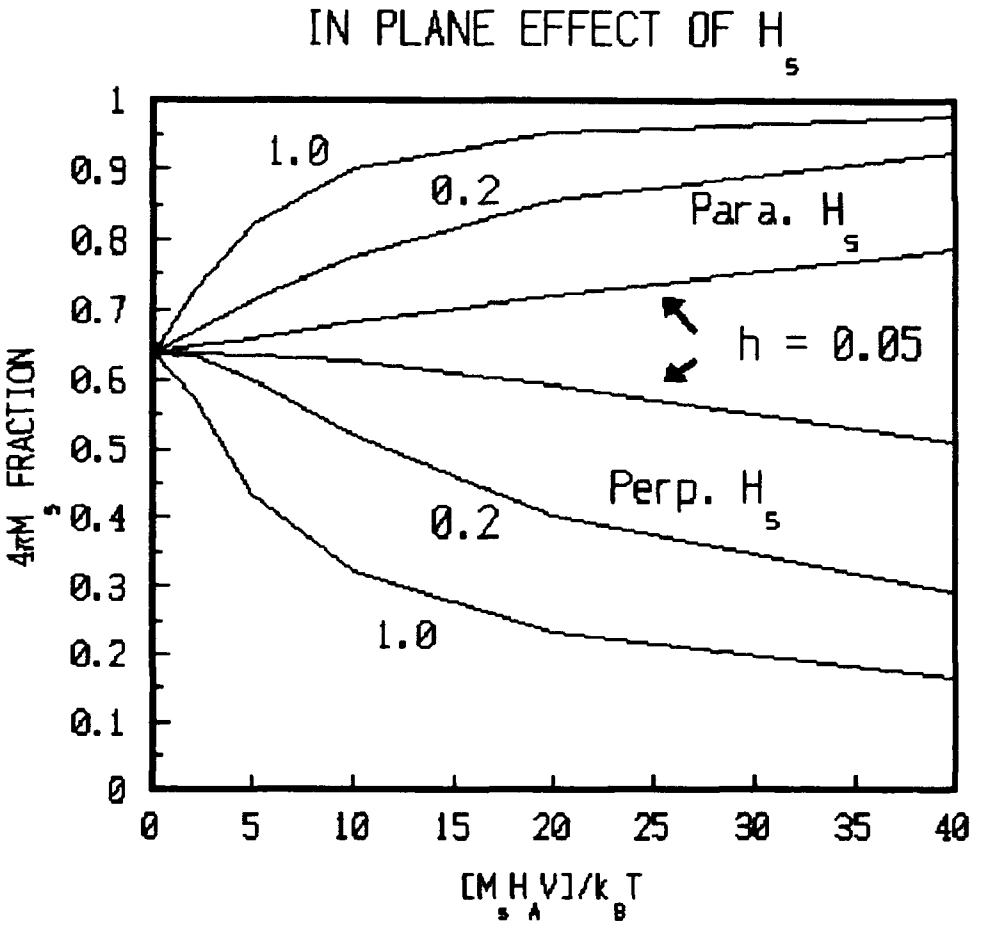

Fig. 6. The calculated expected ratios $4\pi M_r/4\pi M_s$ following initial saturation for different values of $h = H_S/H_A$ are shown for measurements made in plane parallel and perpendicular to the H_S direction. The model is a relaxation by rotation collection of uniaxial single domain crystallites with c-axes confined to the substrate plane. The integrals were evaluated using Mathcad.

C. Iron Enriched True Rhombohedral 2-17 Films

True rhombohedral 2-17 type $Sm_2(Co,Fe,Cu,Zr)_{17}$ films have also been deposited for films with higher Fe concentrations.[9] In contrast to the

insensitivity to subsequent heat treatments of the $TbCu_7$ type films, films that have been made with an increased Fe content generally exhibit large changes in magnetic properties as a result of post deposition heat treatments. Films with an increased Fe content have been deposited by incorporating segments of pure Fe into slits cut into the sputtering target. Such films have the advantage that the saturation magnetization and remnant magnetization can be increased. A fairly large number of films with an Fe content of 24 to 25 at.% have been deposited. The main crystal structure observed for such Fe enriched films has been the rhombohedral 2-17 type with traces of 1-5 phase which constitutes a boundary layer phase. The usual description is that the 2-17 cells are separated by a boundary layer of 1-5 type phase. This is the normal structure observed for bulk type 2-17 type magnets. Bulk samples, as well as the films crystallizing into this structure, exhibit magnetic properties which are sensitive to the heat treatments following initial formation. The subsequent heat treatments are generally thought necessary to properly segregate the 1-5 phase which acts as pinning centers for the 2-17 main phase. Very highly textured films for Fe enriched $Sm_2(Co,Fe,Cu,Zr)_{17}$ samples have been made with the dominant phase being the rhombohedral 2-17 phase.[9] Logarithmic x-ray displays are necessary to display small peaks from the boundary 1-5 type phase. Films have been made so that the 2-17 (220) x-ray reflection is the dominant mode with intensity ratios of greater than 40:1 for the next observed reflection of the 2-17 (300) family. A diffraction trace for such an Fe-enriched sample is shown in Fig. 7.

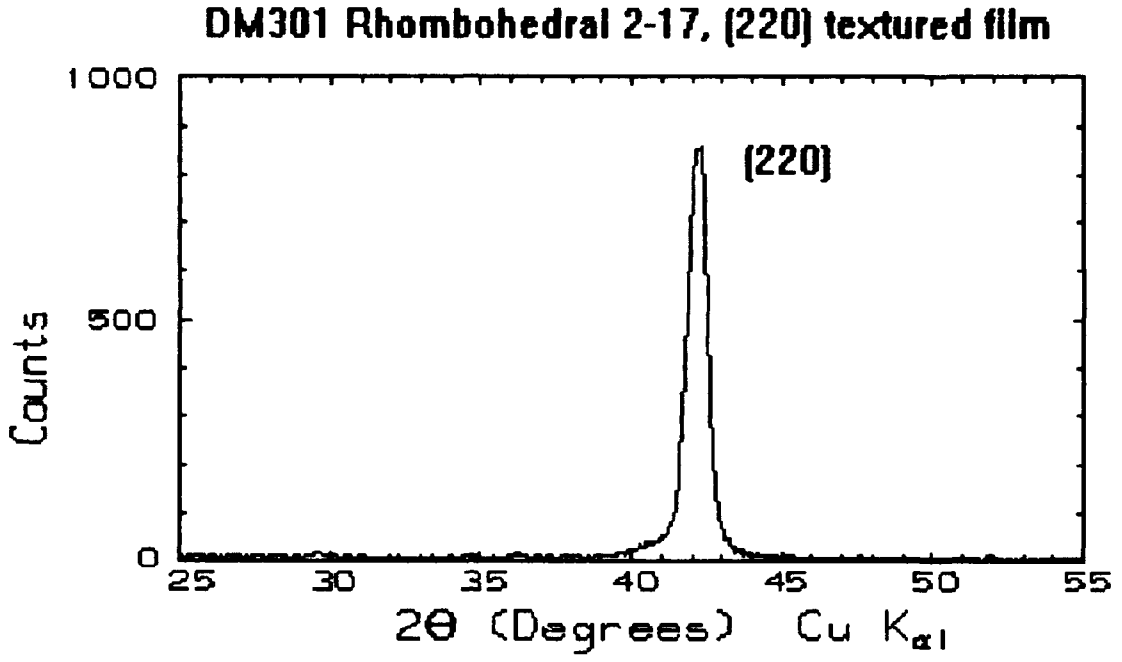

Fig. 7. A $Cu_{K\alpha}$ diffractometer trace for a 2-17 directly crystallized film with (220) texture is shown.

This sample exhibited an in plane energy product of 19.6 MGOe. A hysteresis loop measured in the film plane parallel to H_s is shown in Fig. 8.

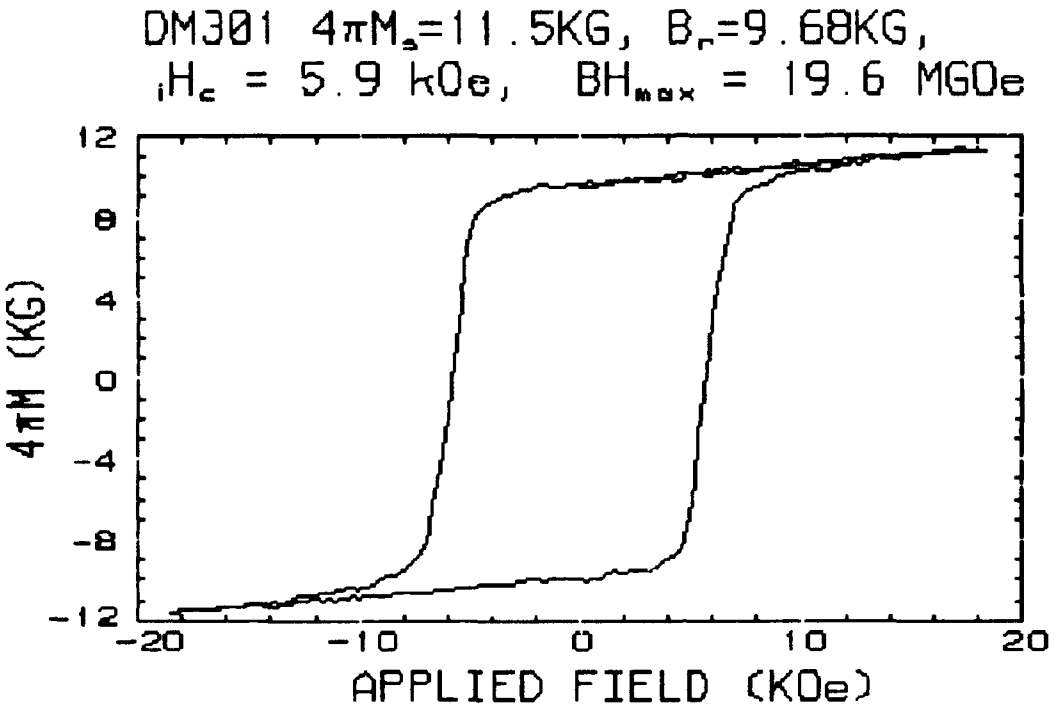

Fig. 8. An in plane hysteresis loop as measured at room temperature is shown for the 2-17 film of Fig. 7. The static energy product was 19.6 MGOe.

An x-ray trace for an Fe-enriched film made at a slightly higher temperature so that the perfect texturing is destroyed is shown in Fig. 9.

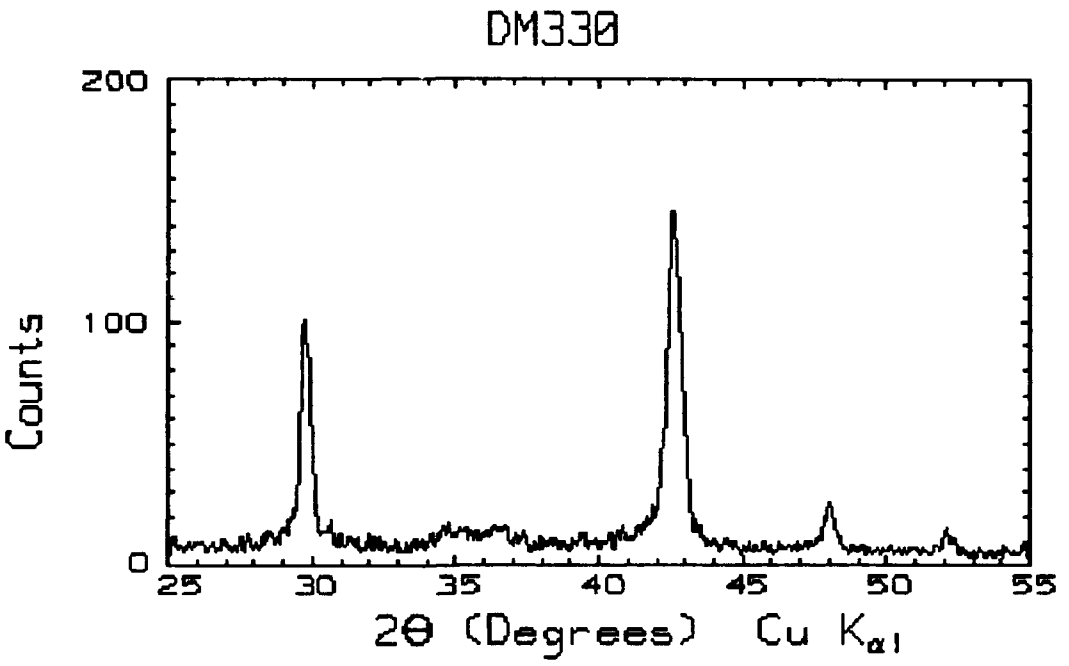

Fig. 9. An x-ray diffractometer trace for a less well textured 2-17 film with a lower energy product is shown. The peaks are: 30.1° = (113), 43.3° = (220), and 48.6° = (223).

These results were presented at the 35th Magnetism and Magnetic Materials Conference held in San Diego, October 1990. A paper related to these results has been published in the Journal of Applied Physics.[9] The high energy product 2-17 films as shown in Fig.'s 7 and 8 have the crystallite c-axes aligned onto the substrate plane within experimental tolerances. Consequently the easy axis of magnetization was also precisely aligned onto the substrate plane. Saturation magnetizations of about 12 kG for $4\pi M$ have been made with remanent values of 10.5 kG. The as sputtered coercivity is reduced however to the order of 2 to 4 kOe. It can be raised to the order of 6 kOe by a subsequent annealing. The sensitivity to subsequent heat treatments is characteristic of cellular 2-17 type systems. Energy products the order of 16 to 20 MGOe can then be realized. The as sputtered films in this case were coercivity limited in their energy products.

D. IMPROVED CRYSTAL TEXTURE CONTROL OF Sm-Co BASED FILMS THROUGH THE USE OF Ar-Xe SPUTTERING GAS MIXTURES

The early synthesis of Sm-Co based film magnets were carried out in Argon. In the direct crystallization of these film magnets, since the thermalization of the sputtered atoms plays an important role, and since the RE and TM components differ widely in atomic masses and radii, the investigation of sputter synthesis of these RE-TM magnet films using mixed sputtering gas species seemed necessary.[1,2,3] A mixed sputtering gas of Ar-Xe can be used to thermalize with high efficiency a sputtered beam of rare earth transition metal atoms. RE-Xe collisions can be used to efficiently thermalize the heavy mass RE atoms at a far lower pressure than could be done by Ar alone. In addition TM-Xe collisions have a large scattering angle for the TM atoms. The RE to TM ratio can thus be varied by varying the total pressure as well as the Xe partial pressure.[3,10] This systematic study of the film property variations with sputtering gas species and other sputter deposition conditions constituted the major part of the thesis of Dr. Hari Hegde.[10]

Through optimized thermalized sputtering very strong crystal texturing was achieved, wherein all the crystallites in the film had their c-axes, that is, their magnetic easy axes, aligned in the film plane. This was indicated by a very narrow hysteresis loop measured perpendicular to the film plane and was confirmed by the x-ray diffraction traces which showed a total absence of reflections from crystallographic planes with nonzero l indices. To a lesser extent, anisotropy within the film plane was also achieved by the application of a magnetic field along a particular direction in the substrate plane. The effect of the field, which we term H_s, was quite significant in films synthesized under optimum conditions of thermalized sputtering. For such films, the energy products for magnetization along the H_s direction was higher, than that for magnetization within the film plane in

the perpendicular direction, by up to 20%. Along H_S, loop squareness, $4\pi M_r/4\pi M_S$, of up to 0.75 have been achieved. It is not possible to achieve higher values because the films of this type are composed of crystallites which have their c-axes aligned onto the film plane, but close to an isotropic distribution within the film plane. Relaxation by rotation of single domain uniaxial particles with c-axes randomly distributed in the film plane, would yield a loop squareness of $4\pi M_r/4\pi M_S = 2/\pi = 0.637$.

The sputtering parameters were also adjusted to make the films sufficiently space filling to the extent that their densities were at least 93.5% of their x-ray density. It has been possible to make highly textured films so that no discernible columnar structure can be detected in SEM cross sections of the films. Thermalized sputtering using 50%Ar50%Xe allowed us to achieve a high degree of anisotropy along the H_S direction at lower Sm concentrations than in films synthesized using Ar only. The remanent induction increases resulting from the decrease of Sm content caused the energy products of these films to be higher than those of films synthesized using Ar by up to 20%. For films synthesized through optimized thermalized sputtering in 50%Ar50%Xe, the maximum energy products were in the vicinity of 20 MGOe for in the film plane measurements.

An attempt was made to explain some of the observed effects, such as crystal texturing, loop squareness and coercivity variation, in terms of a model dealing with the sputtered atom kinematics.[3,5,10] Originally, Professor Cadieu had used such a model to explain the observed high T_c of superconducting Nb_3Ge films, synthesized through RF glow discharge sputter deposition.[11] The model calculates the change in the energy of the sputtered atoms as they undergo collisions with the sputtering gas neutrals and ultimately arrive at the substrate. The calculations of this model differ from the original model of Professor Cadieu, in that, an energy dependent scattering cross section has been used. In addition, this model calculates the change in the energy spectrum of the sputtered atoms as they traverse

from the target to the substrate. The observed changes in the properties of the films have been correlated to the sputter deposition conditions through this model. The model confirms the experimental finding that thermalized sputtering can be carried out either at 60 mTorr of 50%Ar50%Xe, or at 130 mTorr of Ar. According to the model, at these pressures, atoms sputter ejected with even 100 eV of energy would have energies around 1 eV as they arrive at the substrate. This is in conformity with the experimental evidence that such films showed no indication of disruptions caused by energetic massive particles that result in nonlinear demagnetization behavior and that in such films the c-axes of the grains were all aligned in the film plane.

Figure 10 shows the calculated distribution for the sputtered atom energies as the sputtered beam advances toward the substrate. This figure is for the distribution of Co sputtered atom energies using an energy dependent scattering cross section in 60 mTorr Ar-50%Xe. Figure 11 shows the expected energy distribution for Co atoms sputtered in 60 mTorr of Ar only using a constant thermal scattering cross section. Experimentally 60 mTorr of Ar-50%Xe is required to synthesize fully textured films. This is only in agreement with the energy dependent scattering cross section model.

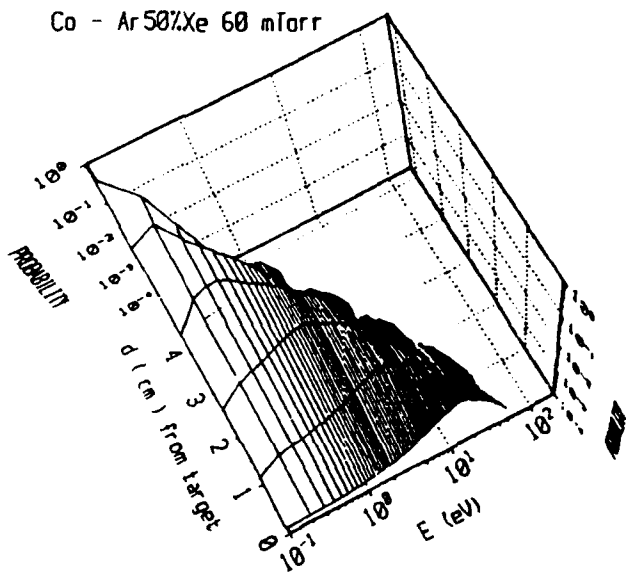

Fig. 10. The energy probability distribution for Co atoms sputtered in 60 mTorr Ar-50%Xe is illustrated as a function of the distance from the sputtering target. A planar geometry has been used. The most usual target to substrate distance is 5 cm. Energy-dependent scattering cross sections have been used. A sputtering gas temperature of 800 K was used in the calculations.

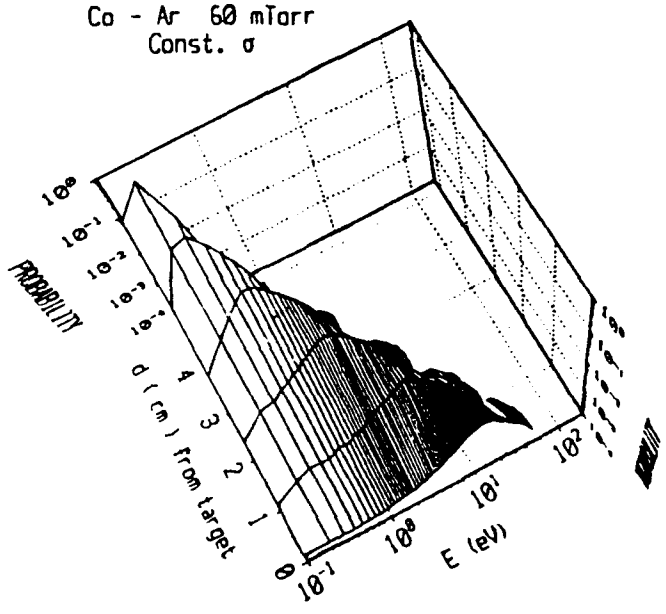

Fig. 11. The expected thermalization of Co atoms sputtered in 60 mTorr Ar based on energy independent cross sections is shown. The possible thermalization is overestimated by this model, and in actual practice higher pressures are required to thermalize the sputtered atoms.

E. DEPOSITION OF FILMS WITH HIGH REMANENT MOMENT PERPENDICULAR TO THE FILM PLANE

Until very recently it has been difficult to make high energy product films such that the magnetization can be directed perpendicular to the film plane. Now we have developed methods that allow Fe rich $SmFe_{12-x}T_x$ films crystallized into the $ThMn_{12}$ crystal structure to be synthesized with very pronounced crystallite texturing.[4,12] The most important texture mode is the almost totally (002) textured $ThMn_{12}$ mode. For this mode the c-axes of the crystallites are oriented perpendicular to the film plane. For this case it is possible to make films so the remanent flux density can very nearly equal the saturation value.

Films that exhibit large remanent moments perpendicular to the film plane have been deposited from Nd-Fe-B type targets by sputtering at fairly low rates so that the growth of crystallites with c-axes aligned parallel to the substrate plane is disrupted. Films of this tetragonal based system then tend to have the c-axes of the $Nd_2Fe_{14}B$ crystallites preferentially aligned perpendicular to the film plane.[8]

It should be noted that for either Sm-Co based systems or for $Nd_2Fe_{14}B$ that high values of coercivity are required to allow a large remanent moment perpendicular to the film plane at H applied equal zero to be retained . The previous $Nd_2Fe_{14}B$ films mentioned that exhibited 10 kG perpendicular to the film plane had a coercive forces from 16 to 18 kOe.

During the past year we have made a very important breakthrough in that a high energy product system has been discovered that can be synthesized such that the easy axis of magnetization is directed perpendicular to the film plane.[4,12] This new system is of the form $Sm(Fe_{1-x}T_x)_{12}$ with $x < 0.1$. The crystal structure is of the $ThMn_{12}$ type. In bulk it has not been possible to synthesize this system with x values as low as

this, or to make $ThMn_{12}$ bulk samples with aligned crystallites. A sample with a random orientation of crystallites can at most exhibit 25% of the energy product of an aligned sample. In bulk it has not been possible to synthesize $ThMn_{12}$ samples without replacing about 20% of the Fe with a nonmagnetic third element such as Ti. In film form we have been able to make $ThMn_{12}$ type films even when x = 0.0, but a x value of about 0.05 is necessary to obtain sufficient coercivity so that a high energy product can be realized. The central part of room temperature hysteresis loops, measured perpendicular to the film plane, and in the film plane, are shown in Fig. 12. The static energy product measured perpendicular to the plane was 20.7 MGOe.

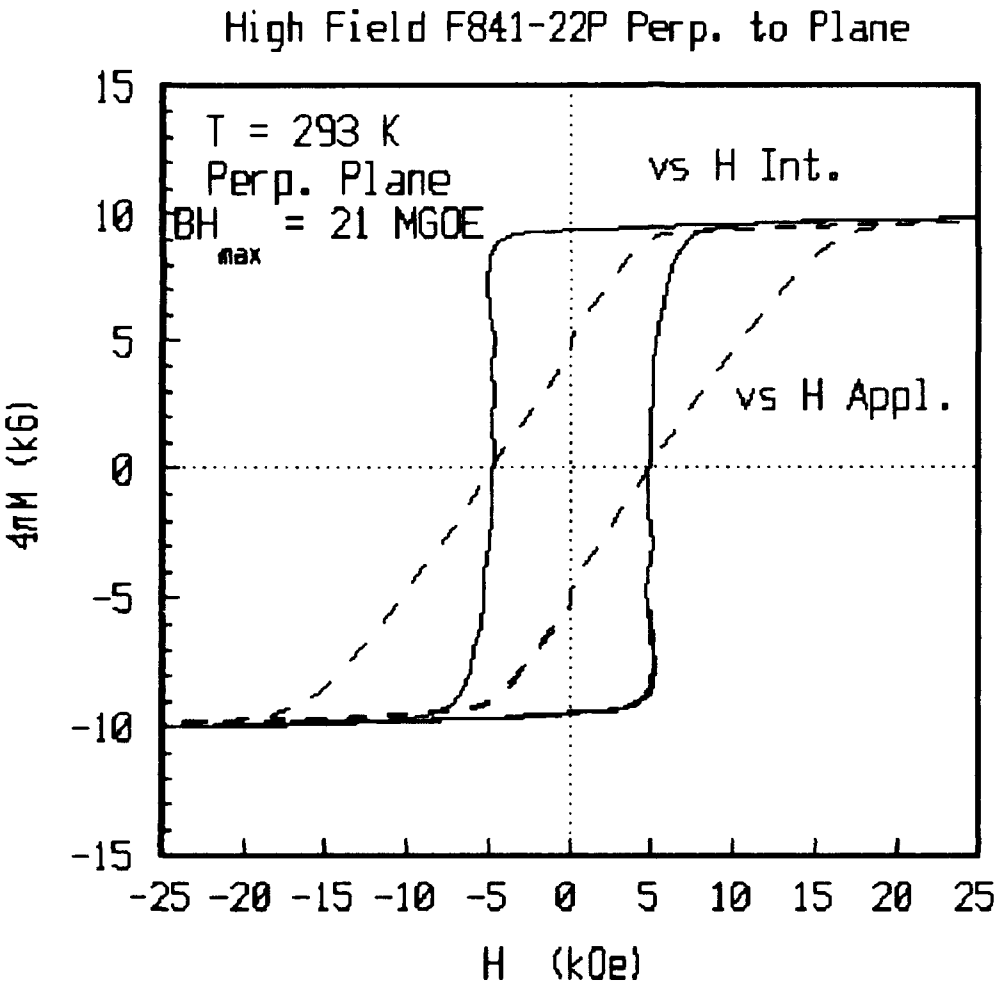

Fig. 12. Room temperature hysteresis loops measured perpendicular to the film plane for a highly textured $Sm(Fe,T)_{12}$ sample are shown. The energy product with **M** directed perpendicular to the film surface was 20.7 MGOe.

High field measurements were necessary to magnetize such samples in the film plane. Hysteresis loops measured to $\pm$90 kOe are shown in Fig. 13. The anisotropy field is estimated to be 130 kOe for this sample.

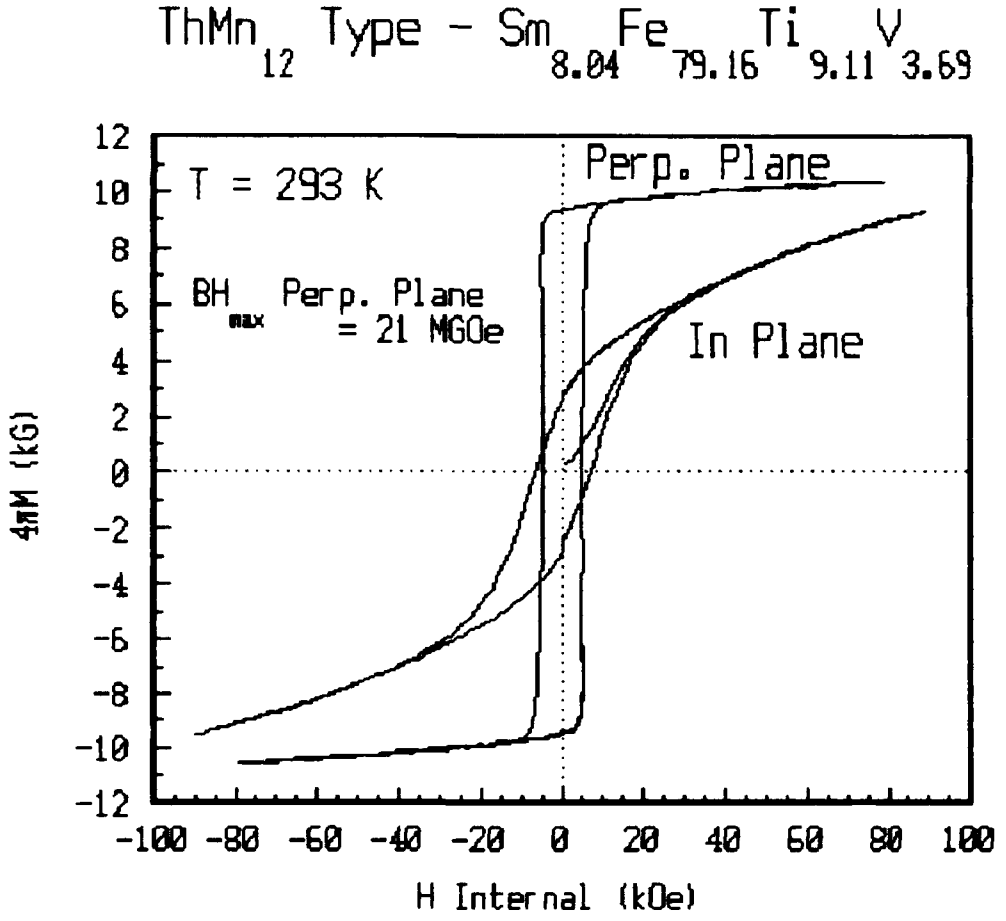

Fig. 13. The room temperature anisotropy field was 130±10 kOe. Since all the c-axes were perpendicular to the film plane, this is a direct measurement of the magnetocrystalline anisotropy field.

III. THE FABRICATION OF MULTILAYER TYPE GEOMETRIES

It is desirable to be able to deposit magnetic films with different types of anisotropies as successive layers. Such layers in proximity can be used to clad films with in the plane anisotropy to direct the flux in useful directions.

An example of the hysteresis loop measured in plane for a film consisting of a high coercivity Sm-Co $TbCu_7$ type layer and a layer of lower coercivity $TbCu_7$ is shown in Fig. 14. This films is such that the

magnetizations in successive layers can be magnetized in opposite directions by the application of a reversed field with a value between that of the coercivities of the low and high coercivity layers.[6,13]

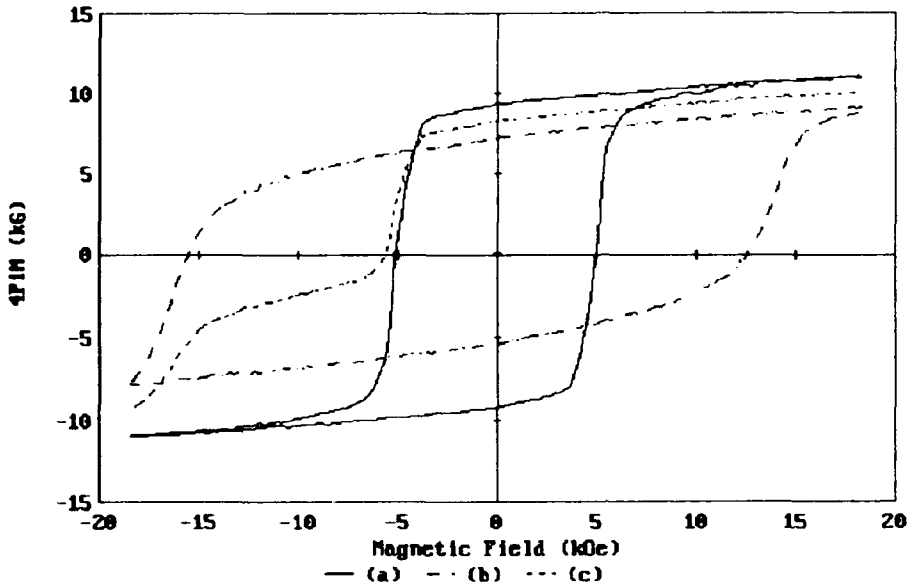

Fig. 14. The hysteresis loop of a lower coercivity $TbCu_7$ type film layer is shown in the solid curve. The alternating layer has a high coercivity of 16 kOe. The demagnetization behavior of the composite layered structure with a stepped pattern is also shown. After initial saturation, a reversed field of about 10 kOe can be used to only reverse the magnetization of the low coercivity layers.

The use of such methods allows miniature film scale periodic permanent magnet arrays to be constructed with spatial periods the order of 0.1 μm to 100 μm.

IV. STRIPLINE GEOMETRY DEVICES, PHOTOFABRICATION OF FILM SCALE MAGNET DEVICES

Recently we have begun to fabricate magnet circuits by photofabrication methods from high energy product magnetic films the order of 35 μm thick. The first devices fabricated have been strip transmission lines such that the strip is situated between the poles of a film patterned magnet. An example geometry is shown in Fig. 15.

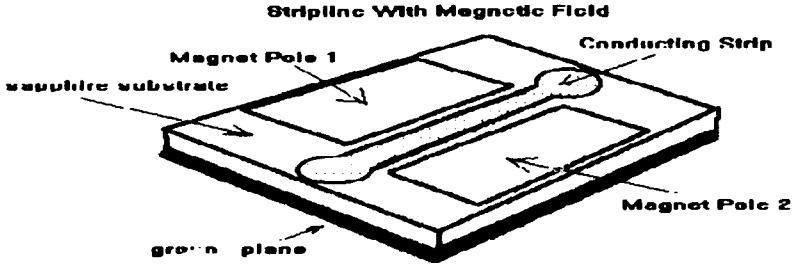

Fig. 15. Relatively thick permanent magnet films, ≈35 μm thick, have been patterned by photolithography methods so that a bias magnetic field can be applied to a conducting strip.

In the figure only the simplest single stripline geometry with the addition of a magnet element is shown. But by photofabrication methods a large number of magnet film patterns can be fabricated onto a single substrate. Meander or zig-zag patterns can readily be used to increase the stripline length. The magnet films used have been directly crystallized Sm-Co based films that have been deposited onto sapphire substrates which have been precoated with a boundary layer. The Al-Al_2O_3 boundary layer has been proved necessary to allow good adhesion of the 35 μm thick permanent magnet films. The films used were synthesized in a magnetic field so that the films were premagnetized as removed from the sputtering system. Thin

film photo resist has been used to pattern the magnetic film. Dilute nitric acid works well to etch the magnetic film. After the magnetic film has been etched away, a sodium hydroxide solution has been used to etch the boundary layer to leave bare sapphire substrate regions. Conducting strips and a ground plane of either Al or Ag are then deposited to provide the microwave transmission elements. Just how fine a line width can be reliably patterned is currently under study. We are not setup to do fine line patterning so that some additional testing will be necessary.

V. GaAs, Si, AND OTHER SUBSTRATE INTEGRATED ELECTRONIC COMPONENTS COMPATIBILITY WITH HIGH ENERGY PRODUCT MAGNETIC FILMS, GaAs, Si, AND OTHER SUBSTRATE CHOICES

For most of the films that we have deposited, the substrates have been either sapphire or fine grained polycrystalline Al_2O_3. These materials are chemically inert for the conditions investigated and stable to very high temperatures. Sapphire has the advantage that it is transparent which can be useful for aligning and the precise patterning of some small scale integrated circuit devices. The use of boundary layers for the deposition of thick magnetic films on either sapphire or polycrystalline Al_2O_3 has been tested.

Some depositions using GaAs and Si substrates have also been done with satisfactory results. The GaAs and Si substrates were precoated with an Al-Al_2O_3 boundary layer by sputtering Al in an oxygen rich RF discharge. Some results for films deposited onto GaAs and silicon substrates are indicated below.

Substrate	$4\pi M_{18}$ (kG)	B_r (kG)	$_iH_c$ (kOe)	$(BH)_{max}$ (MGOe)	thickness µm	Sample ID
GaAs	10.7	8.8	3.6	11.6	6.7	DM264
GaAs	10.9	8.5	5.0	13.4	4.4	DM262
GaAs	10.6	8.8	4.4	14.0	9.7	DM272
Si	11.6	8.5	4.6	15.4	3.0	DM260

Further work is planned for these and other possible substrate materials, but these results indicate that most of the basic problems have been overcom. A hysteresis loop for a 9.7 µm thick Sm-Co based $TbCu_7$ film deposited onto a precoated GaAs substrate substrate is shown in Fig. 16.

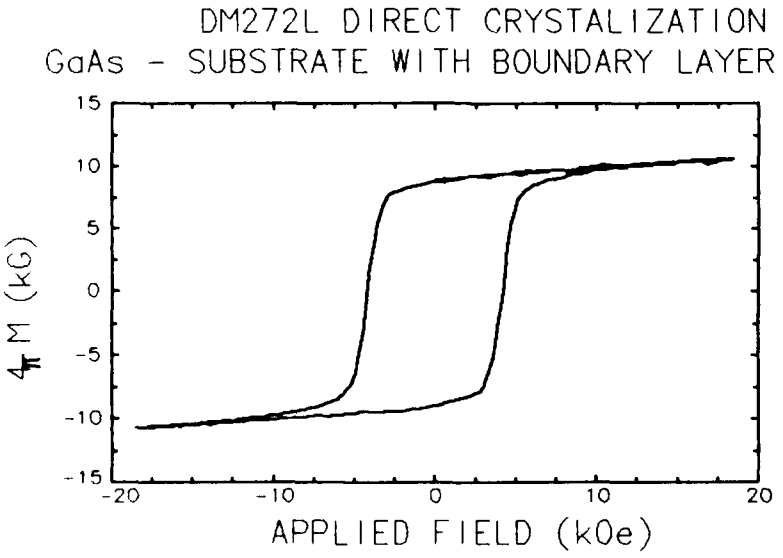

Fig. 16. A room temperature hysteresis loop for a Sm-Co based $TbCu_7$ type film deposited onto a precoated GaAs substrate is shown. This film exhibited a static energy product of 14 MGOe. The film was 9.7 µm thick.

Preliminary work has shown the feasibility of depositing high energy product films onto precoated GaAs and onto Si substrates. Additional experiments are planned to test the compatibility of the deposited magnetic films with the normal electronic circuits deposited onto GaAs substrates. Since we have shown that substrate temperatures for the GaAs during the deposition of the magnetic films only need to be about 400 °C, there is a reasonable expectation that magnetic film components can be directly incorporated into the integrated circuit fabrication. Since Si integrated circuit processing usually employs higher temperatures than used for GaAs, the magnetic films are expected to be compatible with Si chip manufacture.

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SCIENTIFIC PERSONNEL

Professor F. J. Cadieu, Principal Investigator, Department of Physics, Queens College of CUNY, Flushing, NY 11367.

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